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The evolution of damage and strategies for assessing damage when it has occurred in structural materials is the primary focus of this research program. The research effort is collaborative one on mesomechanics called "Damage in 2D and 3D Microsturctures" (AFOSR-89-0423). What distinguishes this effort from others is the attempt to perform incisive model experiments to assist in evaluating stochastic models for constitutive laws (elasticity and conductivity) mechanical response plasticity and hardness and associated damage processes. The theoretical models are designed to be elegant and efficient in the use of computing resources as opposed to brute force procedures which handle systems with different scales by scaling up the size of the computer. The essence of this work is to explore sample size - microstructure relationships via stochastic models which reflect the same types of variability present in the response of real materials. This leads directly to concerns with effective properties of materials. Thus, research integrates the effects of microstructure and preferred orientation on the effective tensorial properties of polycrystalline materials.

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ABSTRACT

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I. INTRODUCTION

1.1. Research on Probabilistic Mesomechanics

In the last several years many investigations have been focused on relating fracture behavior models to experimental fracture data (e.g. 1,2). A major limitation of such efforts is an inability to obtain a satisfactory description of microstructural features which can be related to specific forms of mechanical behavior data. In exploring the mechanics and materials literature, it is easy to recognize the inadequacies of microstructural accounting prevalent in the mechanics literature and the inefficiencies of mechanical behavior representations given in the materials literature. Besides a better understanding of our respective fields, we have succeeded in improving the approaches used for representing microstructures in mechanical models and defining experimental conditions under which models for mechanical response which include microstructural features can be elucidated. The only possibility for better understanding damage processes is to be able to conveniently describe the materials as damage evolves and to resolve the damage as it takes place.

We have been successful in extending models for mechanical response of materials which employ microstructure-based physical representations to describe the mechanical response of materials [3-18]. Mathematical modeling has been advanced into descriptions of intergranular brittle fracture, effective elastic constants and bulk plastic deformation of polycrystalline materials. All of these models rely on efficient mathematical procedures for representation of stochastic variables. Brute force techniques which employ recursive use of random number generators to mimic stochastic variability of mechanical response have been avoided whenever possible. Microstructural features such as grain size and

morphology, crystal anisotropy, and preferred orientation of crystals in polycrystalline materials or composites have been incorporated into the approaches utilized in this investigation. Further information on the first phase of this research are given in Section 1.2.

The essential objective in future phases of this investigation will be exploration of ways to bring together the modeling and experimental work with respect to the original goals of establishing frameworks for modeling and verifying models for damage evolution. As reviewed later, defining stochastic models with features which can be quantified via experimental investigations is a major challenge. To succeed in this endeavor it is necessary to tie the micro- and nanostructural level concerns of materials science to new approaches in mechanics for description of macrostructural performance. The development of analysis tools, both theoretical and experimental, is essential for evaluating the success of the models. Our extension of the research into the measurement of effective properties and property anisotropy, including electrical, thermal and mechanical properties enables us to predictively determine these properties for a given microstructure in undamaged materials and also predict the changes in these properties as damage takes place. The only way in which we can possibly understand the new classes of composite materials is via development of tools which measure quantifiable information. In the next section our accomplishments in the initial funding phase are reviewed, followed by a description of our plans for the next phase of the investigation.

1.2. Accomplishments of the First Phase: The Application of Mesomechanics to "Damage in 2D and 3D Materials"

The global research effort during the first phase has focused on the long range goals:

- **Develop** a microstructure-based approach towards damage mechanics in materials.

- **Tie** the micro- and nanostructural level concerns of materials science to new approaches in mechanics for description of macrostructural performance.

- **Test** stochastic damage models for two-dimensional (2-D) microstructures [5] via experiments on model specimens. Subsequently, the experience gained in the merging of the two-dimensional experimental and theoretical programs will be applied in the extension to three-dimensional materials.

- **Anticipate** the needs of the extension to 3-D microstructures by simultaneously developing the analysis tools, both theoretical and experimental, which are essential for evaluating the success of the models. This is the primary motivation for work on effective moduli and wave propagation in multiphase (structural phases in addition to damage states) materials [12].

We have attempted to create a unified experimental and theoretical program to assess types of damage which occur in polycrystalline materials. We have successfully demonstrated the production of two-dimensional microstructures in materials systems where alteration of the damage characteristics is possible by embrittling initially ductile materials [3,4,19]. The continuation of this work will focus on providing an accurate description of damage and failure in such materials. For these materials will then can employ the recently developed theoretical models which have similar characteristics to evaluate damage and failure processes.

The theoretical basis for this work requires the recognition that the conventional deterministic continuum mechanics is unsuited to the spatio-

temporal random processes of damage. Thus, a graph model (Fig. 1a) of a microstructure plays a fundamental role for the spatio-temporal description of damage phenomena at the microscale -- i.e., on a system of nearest and further grain boundaries -- as well as for making a passage from a finite aggregate of crystals to a *meso-continuum.* In the first case, we have a framework for grasping the microscale interaction effects (e.g. clustering of cracks), while in the second, we have a natural means for relating the size effects in effective continuum-type constitutive laws to the intrinsic microstructural randomness. Here the constitutive response may be of elastic (Section 2.2), viscoelastic, thermoelastic, plastic, and damage-type (Section 2.3), and so, our research provides a first-ever unified framework for derivation of statistical *meso-continuum models.* As discussed in Sections 2.2.3 and 2.3.4, these results are crucial in developing probabilistic versions of finite element and difference methods correctly based on micromechanics. We must point here out that probabilistic finite elements under developments since the early eighties were lacking such a basis.

The approach summarized above provides the opportunity to evaluate the damage process and damage mechanisms in functionally two-dimensional materials. To address the problem of three-dimensional material response it is essential that we explore effective properties of materials and how they might change with changes in microstructural features. Since almost all direct measurements of microstructural characteristics are by nature destructive, particularly microcracking and pore evolution as might be present in a damaged material, concurrent development of analysis via models and nondestructive measurements could be very important with respect to inspection and design considerations. By bringing together experimental measurements on properties and property anisotropy in random and non-random microstructures we can

provide a framework for predicting properties and property anisotropy as damage occurs. Thus far in our research a number of models have been developed and conductivity and elasticity measurements are underway for several instances.

More than fifteen publications from this research already appeared in print. Within the thesis research of current and future students we anticipate continued good productivity in the continuation of this research program.

2. RESEARCH PROGRAM

2.1. Theoretical Analysis

Our research so far focuses on two-dimensional (2-D) materials. The graph representations introduced by Ostoja provide a convenient format for relating theoretical models of two-dimensional microstructures as shown in Figure 1.

The primary reasons being:

(i) a very convenient setting for measurements of damage phenomena and development of a micromechanical theory (discussed below in Section 1.2).

(ii) a testbed for study of many 2-D engineering materials. However, to test functionally two-dimensional materials [3,4] the formation of two-dimensional microstructures within materials which can be processed to produce intergranular fracture is necessary.

A fundamental role in our formulation is played by the concept of a *random medium*, which, as is commonly done in stochastic mechanics (see e.g. [20,21]), is taken as a family $\mathbf{B} = \{\mathbf{B}(\omega); \omega \varepsilon \Omega\}$ of deterministic media $\mathbf{B}(\omega)$, where ω indicates one specimen, and Ω is an underlying sample (probability) space. Formally, Ω is equipped with a σ-algebra F and a probability distribution P. In an experimental setting P may be specified by a set of stereological measurements, while in a theoretical setting P is usually specified by a chosen model of a microstructure (e.g. a Voronoi model, a white-noise random field model, a layered composite). Ideally, one wants to match P in the latter case with that in the former one. As a generic model applicable to a wide range of different microstructures we take a *graph model* G(V,E), where V is the set of vertices and E is the set of edges connecting them, see Fig. 1a). Properties of the body $\mathbf{B}$ are described by a random field parametrized by the spatial

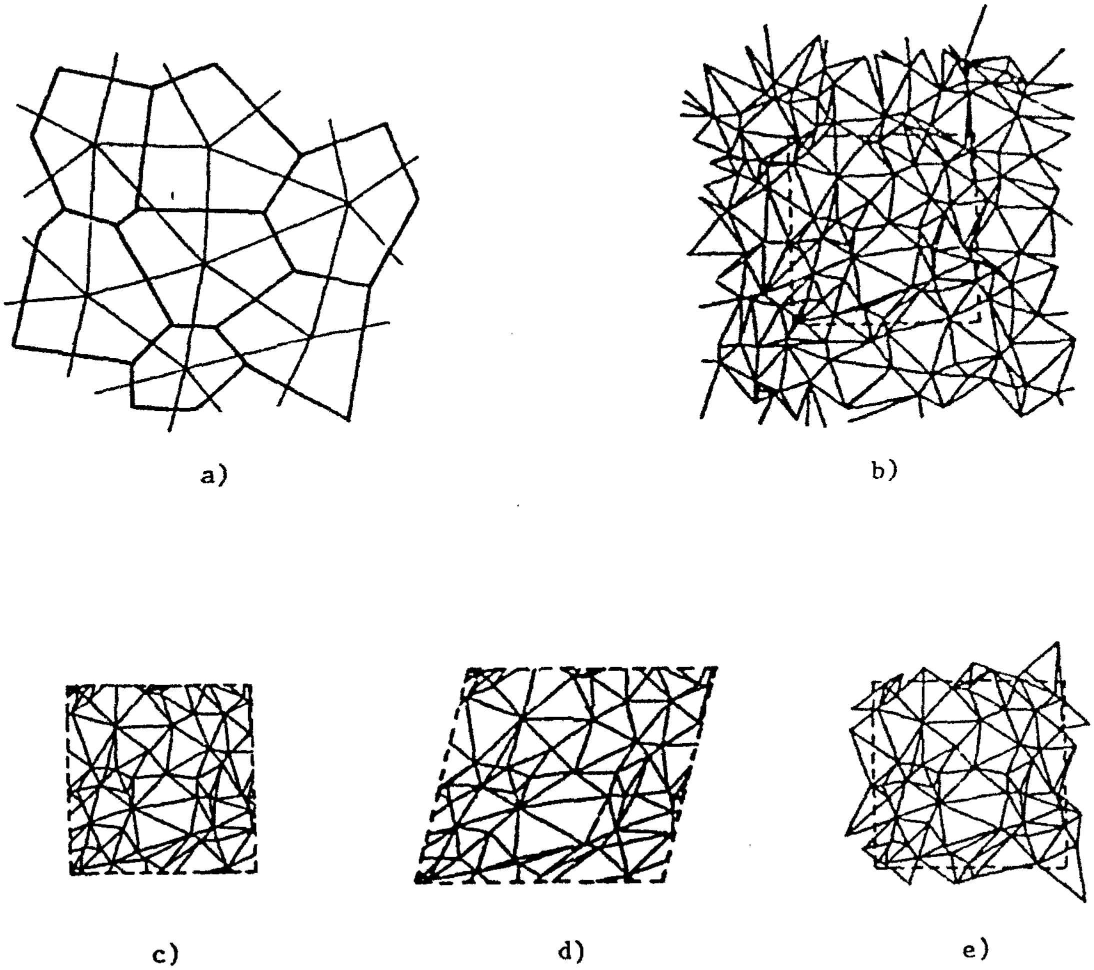

Fig. 1. Graph representation of a microstructure: graph G(V,E) - thin lines, graph G'(V',E') - thick lines.

coordinates and possibly time. In the following we briefly discuss various areas of micromechanics where the above graph formulation plays a basic role.

2.2. Linear Elasticity of Planar Central-force Delaunay Networks (see also Section 2.5)

2.2.1. Hierarchies of Upper and Lower Bounds and Random Continuum Approximations

As a graph G(V,E) we adopt a so-called Delaunay network D(Φ), which is dual to a Voronoi planar tessellation V(Φ); here Φ is a stationary Poisson process in the R^2 plane. We note here that [14]:

- graph G is characterized by a single microscale,
- all edges representing vertex-vertex interactions are linear elastic springs
- statistics of microscale properties are space-homogeneous and ergodic.

Taking the Delaunay network as a linear elastic truss we obtain a generic model of a medium with central-force interactions and geometric disorder [10,22]. Next, with the help of Fig. 1b) and c), we introduce a window of size

$$\delta = \frac{L}{d} \tag{2.1}$$

Equation (2.1) defines a nondimensional parameter δ specifying the scale L of observation relative to a typical microscale d (e.g. grain size) of the material structure. In view of the fact that the Delaunay network is a result of a specific random point process in plane, the window bounds a random microstructure $\mathcal{B}_\delta = \{\mathbf{B}_\delta(\omega); \omega E \Omega\}$, where $\mathbf{B}_\delta(\omega)$ is a single realization.

In order to define the effective moduli of $\mathbf{B}_\delta$, we introduce two types of boundary conditions on its boundary $\partial\mathbf{B}$:

- deformation-controlled (essential) boundary conditions on ∂B, Fig. 1 c) and d);
- stress-controlled (natural) boundary conditions on $\partial\mathbf{B}$, Fig. 1 e).

It follows now that in a continuum setting the effective stiffness tensor or any specific body $\mathbf{B}_\delta(\omega)$ is either

$$C_\delta^e(\omega) \qquad \text{or} \qquad C_\delta^n(\omega) = (S_\delta^n(\omega))^{-1} \tag{2.2}$$

depending on which type of boundary conditions is used; superscripts ^{e} and ^{n} indicate essential and natural boundary conditions, respectively. It follows from the principles of minimum potential energy and complementary energy that, in the sense of eigenvalues, for every $\delta' < \delta$

$$C^R = (S^R)^{-1} = \langle S_1{}^n\rangle^{-1} \le \langle S_{\delta'}{}^n\rangle^{-1} \le \langle S_\delta{}^n\rangle^{-1} \le C^{eff} \le \langle C_\delta{}^e\rangle \le \langle C_{\delta'}{}^e\rangle$$
$$\le \langle C_1{}^e\rangle = C^V \tag{2.3}$$

where $\underline{C}^{eff}$ denotes the effective stiffness tensor of a deterministic continuum corresponding to the scale $\delta = \infty$, $\underline{C}^V$ is the Voigt bound, $\underline{C}^R$ is the Reuss bound, and denotes ensemble averaging. In fact, one can show that this is a limit $\underline{C}^{hom}$ sought in the homogenization theory [23], in which the parameter ε is analogous to the inverse of our δ. A computational method for determination of the hierarchy (2.3) is discussed in [9,14].

2.2.2. Determination of Effective Moduli via the Self-consistent Method

The problem of determination of effective moduli of Delaunay networks falls into a general category of linear transport problems. Their important subcategory is concerned with the effective medium theories, such as the self-consistent method. There are two basic cases: randomness per edge, or

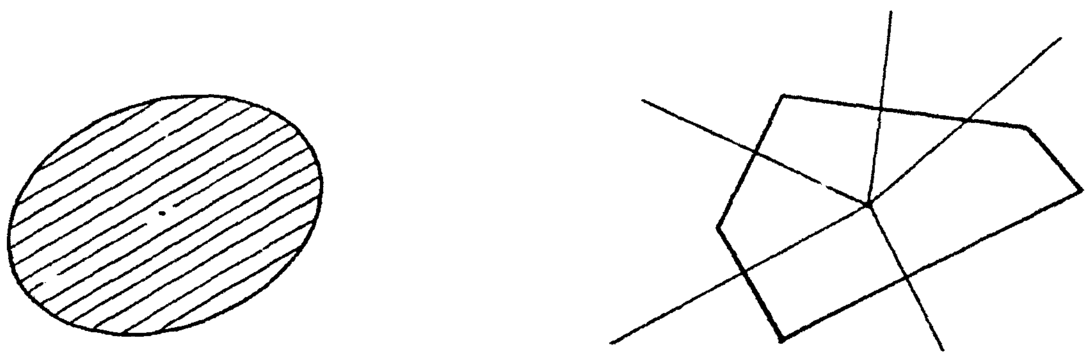

Fig. 2. Representation of a Voronoi Cell Embedded in a Matrix.

randomness per vertex. The first case is easier to handle and has been solved in a number of conductivity and elasticity problems, see e.g. [24]. The second case is especially hard in case of a topologically disordered microstructure lacking periodic (i.e. Bravais lattice) geometry.

Our present research on this problem, is based on a "spider - inclusion" analogy, where a spider connotes the vertex v and those halves of edges incident onto it which lie in this vertex' Voronoi cell. The idea, presented in Figure 2, relies on an observation that the Voronoi cell (or grain) surrounding the given vertex can be thought of as an elliptical-type inclusion imbedded in a matrix. Henceforth, follows the applicability of the self-consistent methods developed in mechanics of polycrystalline materials. The idea is to find the effective moduli of a Delaunay network, i.e. a system of random "spiders" - as if it was a field of inclusions without conducting the computer-intensive calculations of large windows. Various possibilities arise:

- inclusions with perfect or imperfect bonding at with the matrix,
- circular or elliptical inclusions,
- symmetric or asymmetric formulation of a self-consistent scheme,
- Voigt and Reuss bounds for systems with perfectly or imperfectly bonded inclusions. Our results on this subject are reported in [11].

2.2.3. Autocorrelation Functions of the Random Field Approximations

In the theory of stochastic finite elements a fundamental role is played by the autocorrelation functions of the material properties. Definitions $(2.2)_1$ and $(2.2)_2$ are analogous to a moving locally averaged random field (see e.g. [26]), although no direct straightforward averaging is possible, but, rather, computations must be carried out. The normalized *autocorrelation* functions are defined so that the δ-dependence of C_{ijkl}'s is implicit, recall (2.2). In contradistinction to the presently employed procedures - such as the "weighted

integral method" [27], or the "spectral representation method" [28] which have no connection to the material microstructure our approach of the preceding subsection provides a rigorous basis for determining C_{ijkl}'s.

Analyses carried out so far in this vein in [10] for Dealunay networks lead to the following principal conclusions:

i) the autocorrelation functions of shear moduli are isotropic, while those of other moduli are anisotropic,

ii) the uniform strain approximation results in practically identical autocorrelation functions as those obtained by the exact method, and thus suggest a very inexpensive computational method,

iii) the isotropy of material is approached only asymptotically simultaneously with the coefficient of variation tending to zero; thus, the usage of random locally isotropic media models employed by the present practitioners of stochastic finite elements with noise-to-signal ratio of up to 30% is physically unrealistic. In fact, even the assumption of a Gaussian character of, say, Young's modulus is unjustified there (!).

Employing our theoretical approach we are in the process of determination of the probability distributions of effective elastic moduli C_{ijkl}, and their dependence on δ, as well as their autocorrelation functions and higher order moments. However, as it was already pointed out, two alternative definitions of boundary conditions are possible - deformation-controlled and stress-controlled - from which two different random anisotropic continua result. Thus, it follows that a given boundary value problem may then be solved by stochastic finite differences or elements [25,33] to find the lower and upper bounds on response according as $(2.2)_1$ and $(2.2)_2$ are employed. These works represent a first-ever(!) formulation of micromechanically-based stochastic finite difference and element methods.

2.3. Extension to Nonlinear Elastic and Dissipative Responses

2.3.1. Nonlinear Elastic Microstructures

At every scale δ, for a random medium, $\mathbf{B}_\delta$, we can introduce two random functionals: ψ_δ - free energy in the space of velocities, and $\psi_\delta{}^*$ - free energy in the space of stresses. Thus, the theoretical framework is complemented by a Legendre transform [18] obtained under essential or natural boundary conditions. Since these relations are not restricted to linear elastic media, they will be fundamental to our analysis of nonlinear elastic microstructures.

2.3.2. Stochastic Continuum Damage Mechanics (see also Section 2.4)

Concerning the dissipative response, we note that the continuum thermomechanics of solids may be formulated on the basis of two functionals: *free energy* ψ and *dissipation function* ϕ, see e.g. [29-32]. In [18], basic relations of thermodynamic orthogonality of this formulation are generalized to random media. Essentially, two types of conditions - controllable velocities or controllable forces - may be considered, leading in each case to a different average (effective) function ϕ; these provide upper and lower estimates on dissipative response. This leads to a reinterpretation of the Legendre transformations and extremum principles in a random setting. Special cases of homogeneous and quasi-homogeneous dissipation functions are considered in detail. Considering the fact (discussed in papers 1 above) that the continuum formulation is dependent on the size of a Representative Volume Element (RVE) relative to the microscale, our generalization provides the basis for continuum random fields in thermomechanics.

A particular application of this theory appeared in [5], with a cornerstone of distinction between the deterministic character of the so-called "continuum

damage mechanics," developed in the eighties, and the probabilistic nature of fracture and damage phenomena at the microscale. Thus, [5] gives a micromechanical formulation of a *stochastic continuum damage mechanics*, which becomes the conventional deterministic continuum damage mechanics only in the limit of an infintely large RVE. These ideas are pursued in [13,34], and, presently, explicit forms of stochastic constitutive laws are being derived.

2.3.3. Damage Percolation Models

Studies of damage phenomena in polycrystalline, fiber-matrix composite and ceramic solids require a proper description of miroscale effects as well as a correct model at the meso-scale. The latter objective follows the lines of point 1 above in that it recognizes the extreme scale-dependence and random nature of damage phenomena. As an example problem, we studied in [6] damage formation in a polycrystalline layer. The model was set in the framework of a graph representation (see Section 2.2.1. above) with damage confined to the honeycomb network of grain boundaries, and their strengths being described by space-homogeneous statistics. A hierarchy of Markov field models was introduced to grasp the clustering effects in the spread of damage between the first, second, third, ..., neighbors. The simplest case in the hierarchy corresponds to a Bernoulli percolation, which can be easily solved. The higher cases of nontrivial Markov fields may be analyzed via a reduction to a problem of phase transition on a graph.

2.3.4. Plasticity of Spatially Inhomogeneous Materials (see also Section 2.6)

The subject of plasticity of randomly inhomogeneous media was mentioned probably for the first time, but not developed, in [35]. We note here that random

distortions of slip-line patterns from those predicted by deterministic medium theory are frequently observed in experiments, see photographs in [36,37]. Thus, in [67] we undertook a generalization of the classical method of slip-lines (characteristics) of planar flow of perfectly-plastic media to the setting of random media. These media are characterized by space-homogeneous statistics of the yield limit k, whose derivation was outlined on the basis of micromechanics. It was shown that the field equations of the random continuum approximation lead to a *stochastic hyperbolic system*. This system, when stated in a finite difference form, displays a Markov property for the forward evolution. On that basis, two methods of solution of boundary value problems - an exact one and a mean-field one - were outlined through an example of a Cauchy problem; an extension to characteristic boundary value problem was given in [69]. The principal observation is that even for a weak material randomness the stochastic solution may differ qualitatively from that of a homogeneous deterministic medium and have a strong scatter. At this point we also make a reference to [70], reporting a study of the effects of material randomness on the safe radius of a cylinder under normal internal pressure in the limit state. Recently, this study has been extended to internal pressure accompanied by a shear traction.

2.4. Failure of Model Microstructures in Real Materials.

2.4.1. Two-Dimensional Microstructures

Our criteria for the extent of two-dimensionality, as defined in the doctoral work of Michael Grah, include large 'average grain size to specimen thickness ratio', orthogonality of grain boundaries to the plane of the sheet material, and small thickness compared to the two other dimensions of the sheet sample [3,4]. Approximately 2-D microstructures have been produced in 3.25%Si-Fe and pure aluminum sheets through optimized cold-work/anneal schedules followed by mechanical thickness reduction with microstructures as shown in Figure 3. Optimization of preparation technique has resulted in Fe-3.25%Si samples with average grain size to thickness ratios of 6 or more. In addition, deviations of grain boundaries from orthogonality have been limited to an average of around 6°, and specimens with length/width to thickness ratios greater than 1,000 have been produced. Results for aluminum are similar.

This area of research can be closely tied to efforts to represent the orientation relationships between grains, called microtexture and the orientation relationships of these grains with bulk geometry, or macrotexture. Watanabe and coworkers [2], have attempted to predictively assess the fracture behavior of materials, particularly silicon-iron, based on assumed microtexture and macrotexture relationships. The major stumbling block to effective application of these models is that the data on microtexture is very limited and that three-dimensional materials are not convenient for characterization of both the microtexture characteristics and the measurement of fracture response. Both these investigators and microtexture researchers, Brent Adams and coworkers, have expressed interest in evaluating these 2D materials.

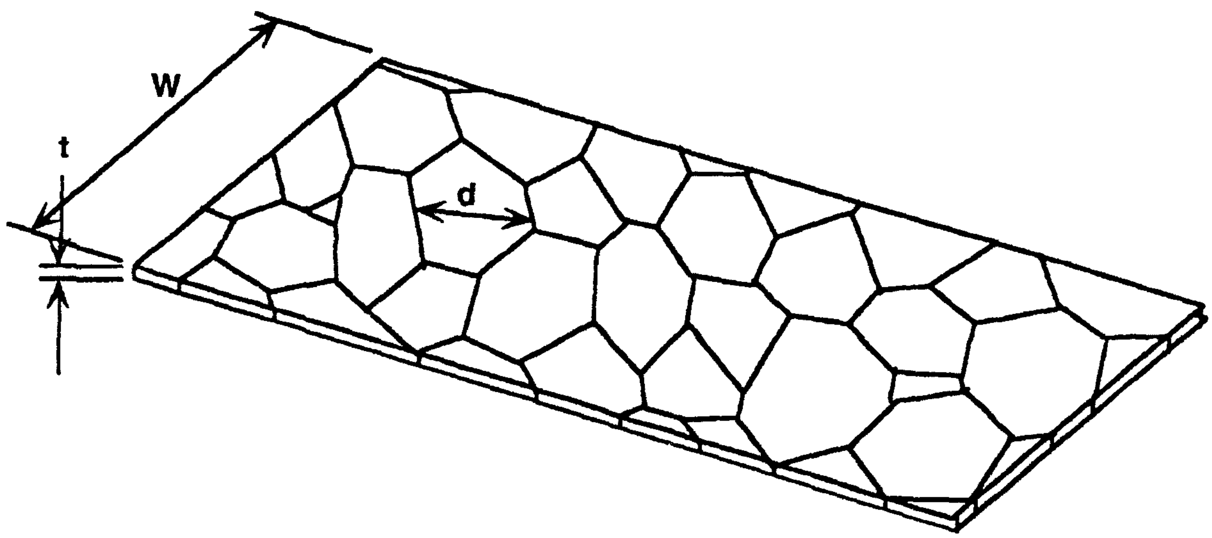

Fig. 3. Schematic of a sheet possessing a two-dimensional microstructure. $d/t >> 1$, $W >> t$, and orthogonal grain boundaries.

We have initiated a collaboration with Drs. Mark Vaudin and Linda Braun of the National Institute of Science and Technology (NIST) to describe the crystal orientations and fracture process in these materials. In this aspect of our research our goal is to find the distribution of certain intercrystalline orientations (grain boundary type), the orientation distribution of the grains and the orientations of specific grains and grain boundaries with respect to the rolling geometry. Once we ascertain this type of information, and develop a convenient format for representing the orientation information, we hope to then relate the orientation information to the fracture resistance of boundaries experiments which will be carried out at NIST and Purdue. By combining the orientation information, the distribution of grain boundary types and the fracture resistance of certain grain boundary types we should be able to describe the stochastics of fracture resistance which should be predicted in the modeli g work.

This research has been extended into work on liquid phase embrittlement of aluminum sheets with two-dimensional microstructures. A schematic diagram of one of these specimens undergoing biaxial fracture is shown in Fig. 4 and micrographs showing crack propagation is given in Fig. 5. Note that the fracture under biaxial loading always proceeds with the three-fold symmetry dictated by the grain triple junctions.

Grain boundary wetting by liquid metals has been shown to cause grain boundary embrittlement in some FCC and BCC metals [38,39]. Specifically, liquid gallium has been shown to cause room temperature grain boundary embrittlement of aluminum, zinc, and other metals [40-44]. Liquid gallium which contacts the metal surface below the oxide film spreads over the surface and through the grain boundaries by wetting. This high spread rate is due to a very low contact angle of 27° for gallium on aluminum and 28° for gallium on

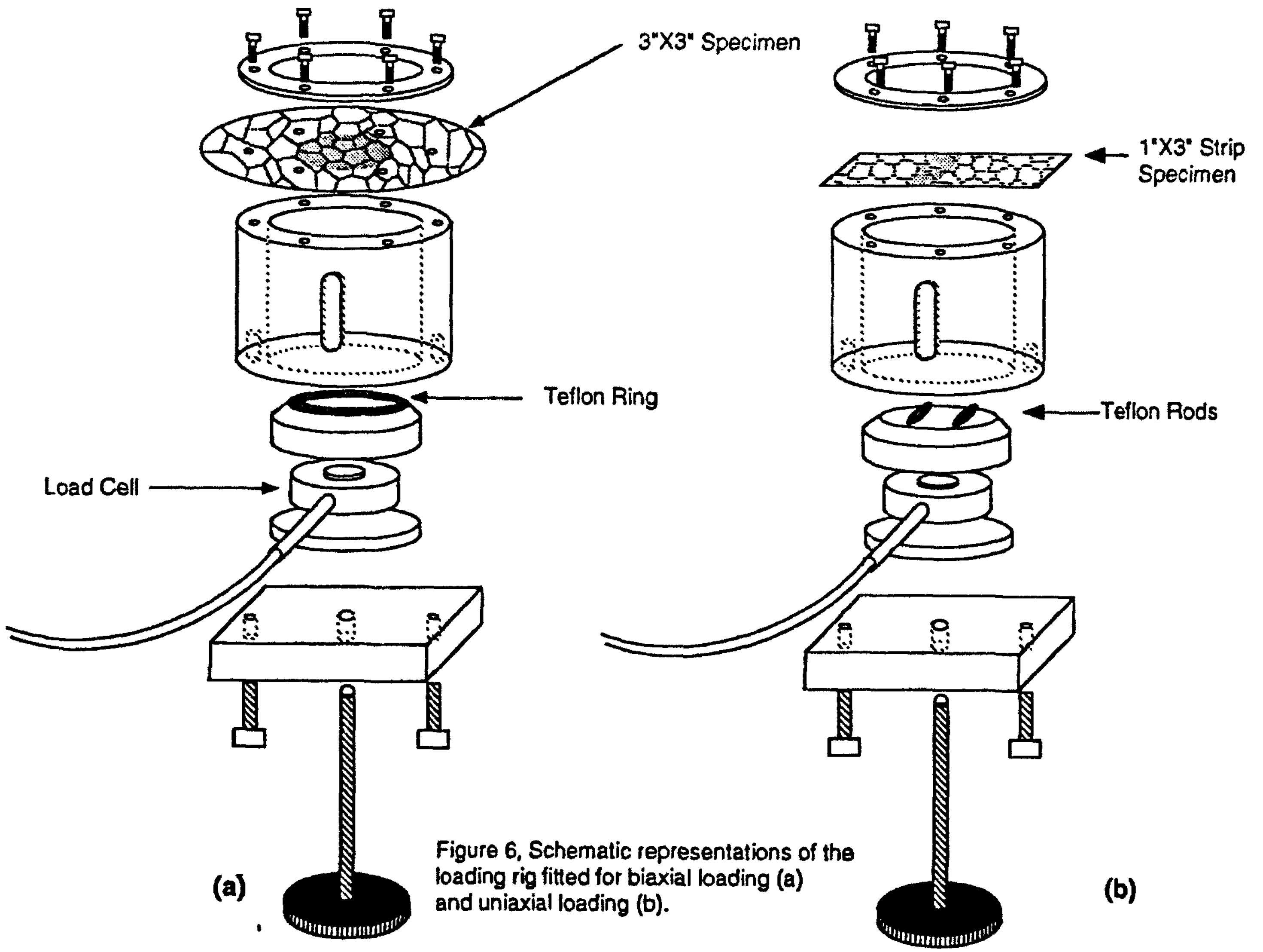

Fig. 4. Schematic representations of the loading rig fitted for biaxial loading (a) and uniaxial loading (b).

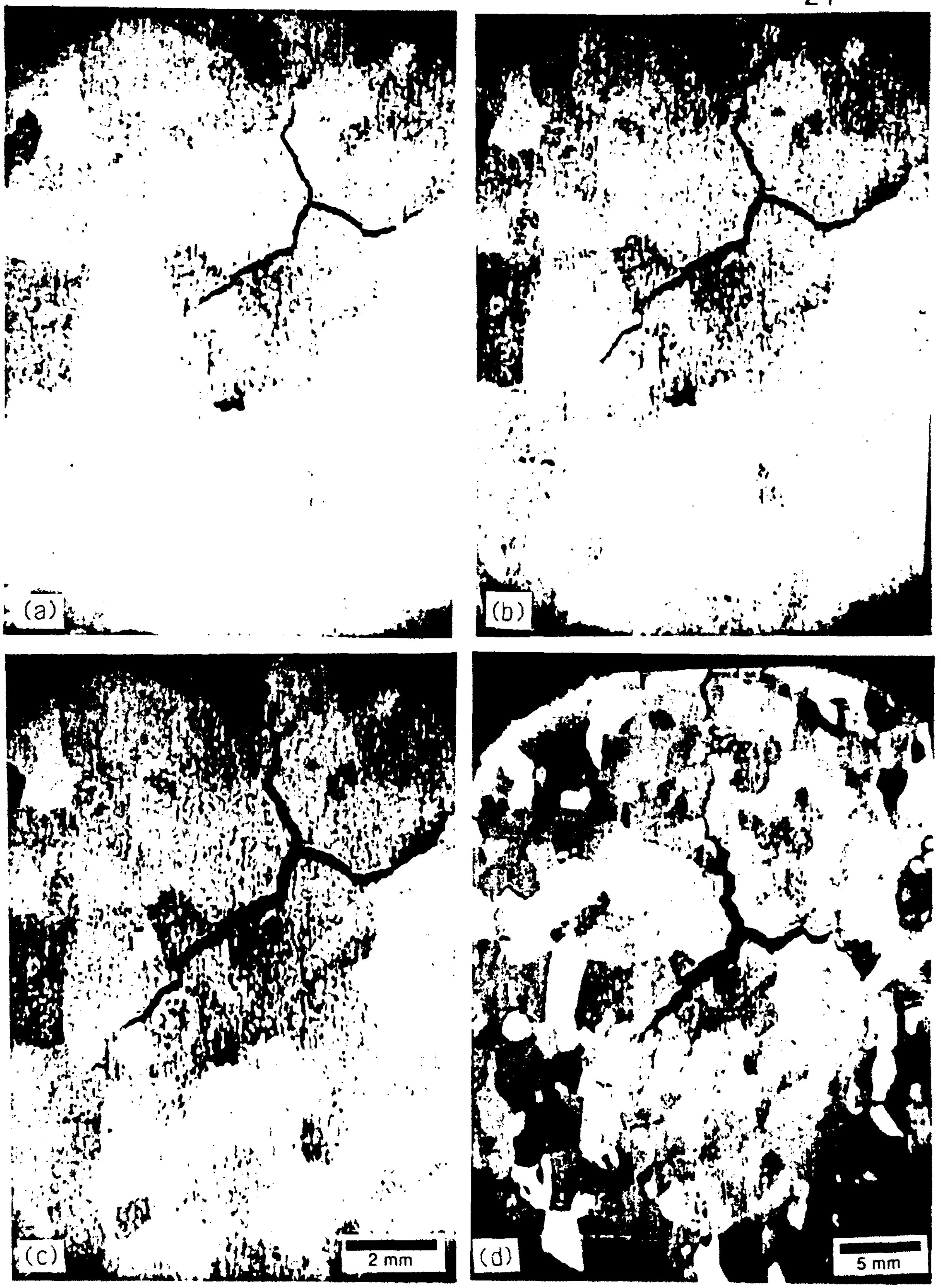

Fig. 5 (a)-(f), Sequential stages in the crack propagation of a biaxially loaded 2D strip.

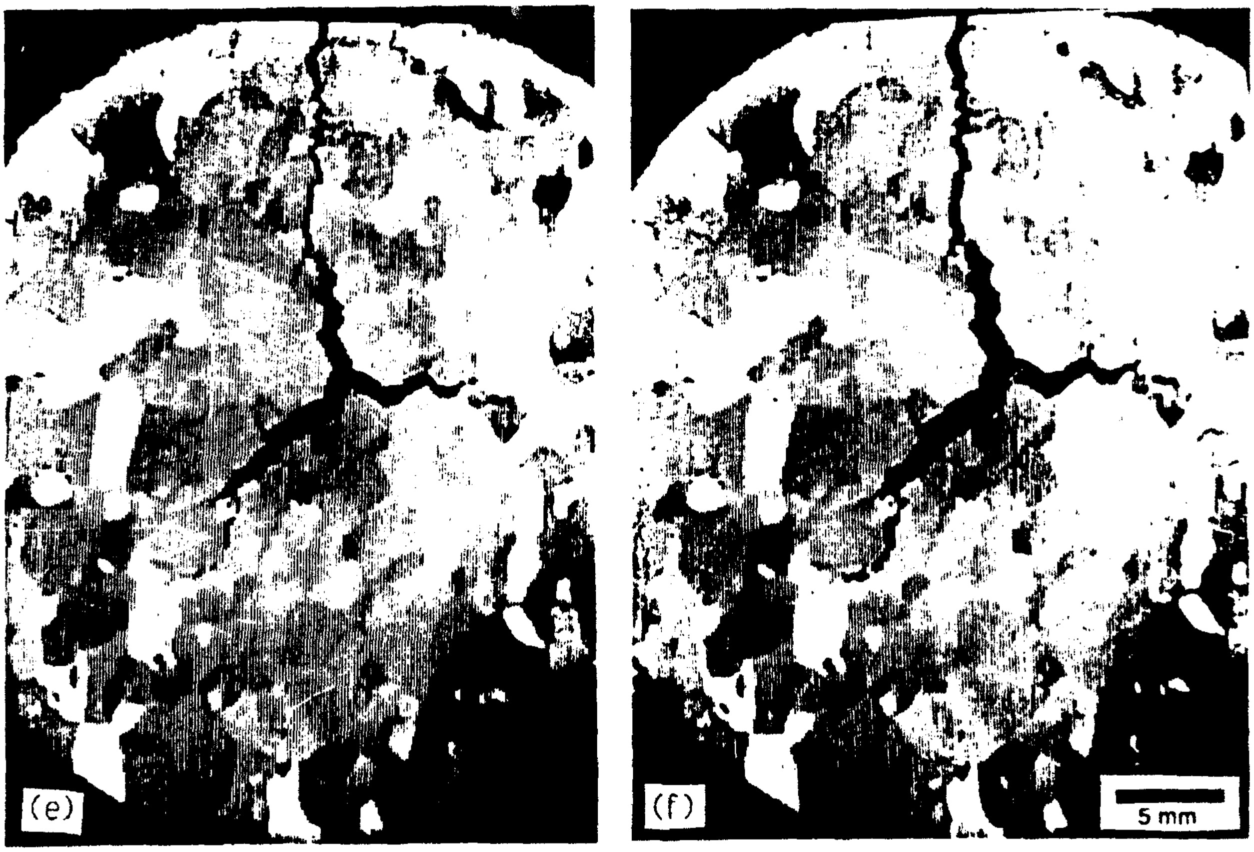

Fig. 5 (Cont.).

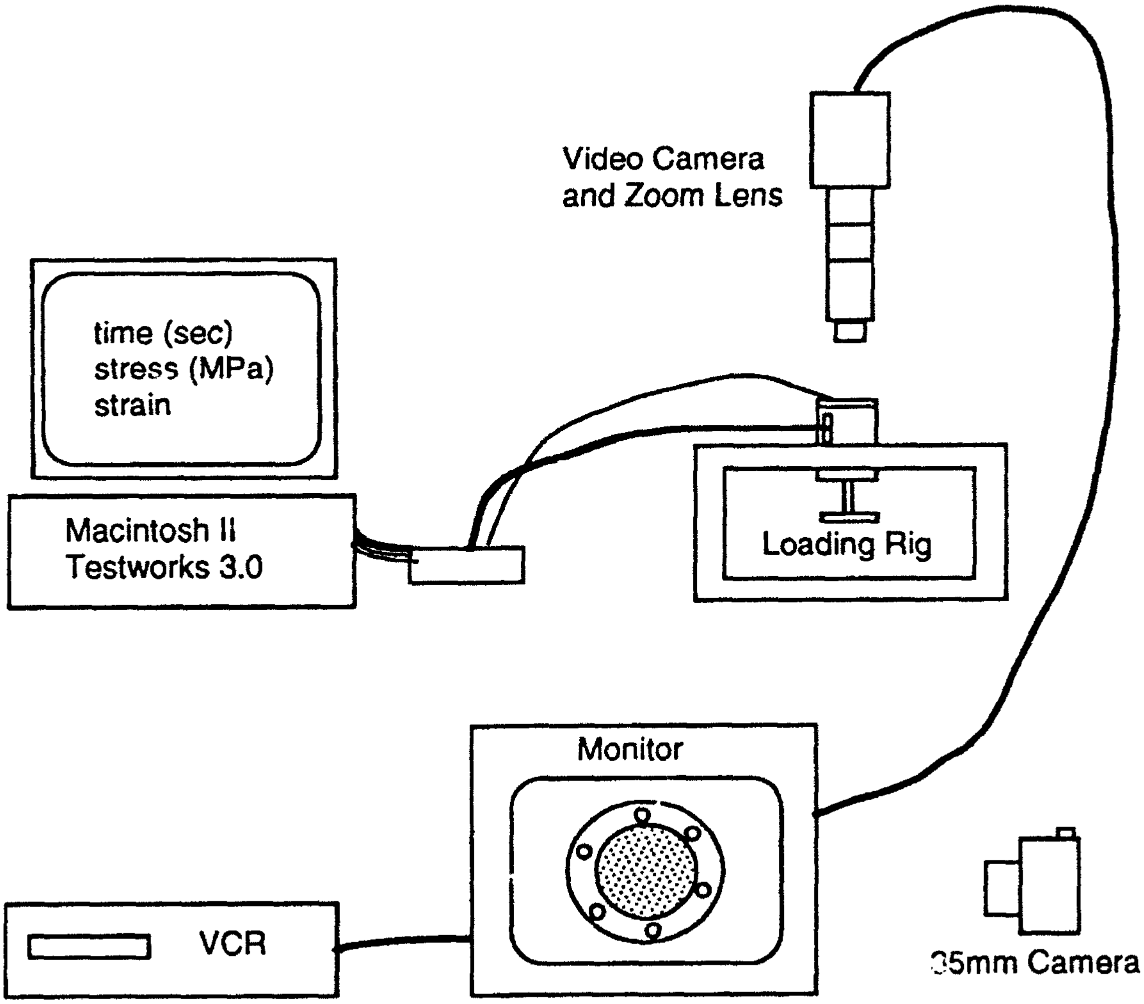

Fig. 6. Schematic representation of the loading rig, measuring apparatus, and recording apparatus.

aluminum oxide [45]. The rate and extent of grain boundary wetting is dependent on the type of boundary being penetrated [40,42]. A thin liquid eutectic film forms on penetrated boundaries and reduces cohesion sufficiently that intergranular fracture occurs before plastic flow under applied stress. The film has been measured to be on the order of 100 Å thick [46,47]. Room temperature fracture of gallium embrittled aluminum is analogous to high temperature creep and fracture of ceramics possessing glassy grain boundary phases.

The objective of this research is to develop a baseline of experimental data for fracture in gallium embrittled aluminum which possesses an approximately 2D microstructure. A method is developed to produce and characterize the extent of two-dimensionality in aluminum strip. An apparatus has been developed (see Fig. 4) to load this material in both uniaxial and biaxial tension in such a way that crack propagation can be easily observed. Techniques have been developed to measure and record applied stress and crack propagation as shown in Fig. 6.

The objective of the embrittlement is to produce a specimen with a 2D microstructure which fails by brittle intergranular crack propagation. The embrittlement techniques were designed simultaneously with the loading rig so that failure could be induced in only the embrittled region. To accomplish this, the gallium is applied so that a controlled area on the sheet was embrittled. When gallium is applied as a dot, it embrittles a disk shaped region approximately 3 cm in diameter. The 1"x3" strips experience embrittlement in a center region approximately 1/2" wide for gallium applied as a 1/4" strip in the center. The area around the embrittled region remains ductile and provides anchor points for the rig and a means to transfer stresses directly to the embrittled area.

At room temperature, aluminum strips embrittled by both methods become profoundly brittle and will not support a resolvable stress on the rig. Also,

fracture of these strips is catastrophic; no observation of crack propagation is possible. Thus, it is necessary to de-embrittle the specimens sufficiently to provide some grain boundary strength. Partially completed systematic studies of de-embrittlement times for both types of gallium embrittlement provide some data necessary to reproducibly produce strips with acceptable fracture properties.

Biaxial specimens embrittled with a dot of gallium were found to have wide fracture strength variability. Apparently, the rates of de-embrittlement are extremely sensitive to the amount of gallium applied. Relatively long de-embrittlement anneals of ≈48 hr were necessary to provide the most desirable fracture properties. In addition, specimens successfully embrittled by this method showed strong increases in the grain boundary fracture toughness with crack length. This R-curve behavior is believed to be due to a gradient in gallium embrittlement from the dot source.

Wetting an entire region on the 1"x3" aluminum strips with gallium was found to yield more consistent fracture results when the excess gallium was wiped off of the surface prior to the de-embrittlement at 95°C. Since only a small amount of gallium was allowed to spread into the internal surfaces, relatively short times were necessary to get the desired amount of boundary brittleness. 1"x3" strip specimens with 2D microstructures required a 1/4" strip of Ga for 15 minutes followed by ≈6 hr. anneal to produce the desired level of embrittlement.

The local embrittlement of the aluminum strips and the nature of the loading rig allow application of stresses where the sheet is ductile and transfer of those stresses directly to the embrittled region. When properly embrittled by the techniques described above, fracture initiates and propagates along grain boundaries. The de-embrittling times were adjusted in an attempt to provide specimens where failure initiates at a remote stress of 10-20 MPa. This extent of embrittlement is chosen because considerable elastic and some plastic strain

occurs prior to fracture. This strain makes observation of the cracks possible. Also, at this level of embrittlement cracks usually propagate slowly enough to observe and record their behavior.

Biaxially fractured specimens showed little indication of plastic deformation prior to fracture. Like the uniaxial strips, the crack path was almost entirely intergranular with the crack faces normal to the plane of the sheet. In testing the biaxial sheets, fracture was observed to consistently initiate at a triaxial junction. Multiple crack initiation was rarely observed. This is in contrast to the uniaxial specimens which experienced fracture initiation along several grain boundary facets oriented perpendicular to the stress axis. Cracks were observed to propagate from the three failed grain boundaries of the triaxial junction and maintain paths approximately 120° apart. Occasional crack branching was observed in the biaxial specimens when a propagating crack approached a junction where both possible crack paths deviated from the main crack path by an angle of greater than approximately 45°. Prolific crack kinking is observed during propagation due to the effects of 2D grain boundaries on intergranular fracture. All the biaxial specimens exhibited strong R-curve behavior. This is attributed to the dot method of embrittlement which develops a gradient in the extent of embrittlement from the dot source. Thus, the grain boundary fracture toughness apparently increases with distance from the original gallium dot.

Two established toughening mechanisms were observed in both uniaxial and biaxial strip specimens. Obviously, cracking kinking was dominant during fracture due to the 2D nature of the grain boundaries. Occasionally, crack bridging was also observed. In these materials, crack bridging is typified by the fracture surface switching from brittle intergranular fracture to ductile tearing with extensive plastic flow on both sides of the bridging grain. These effects are quite visible in the SEM micrograph of that bridging grain shown in Figure 7.

Bridging grains develop when their boundaries were not embrittled as much as those of the surrounding grains. These less embrittled boundaries probably have special properties which make them less susceptible to gallium embrittlement. Boundaries of a low CSL type or low angle boundaries have been shown to exhibit this kind of behavior [40-42]. Recent surface diffraction measurements carried out on similarly prepared strip specimens by M. Vaudin at NIST indicate that a strong 110 texture exists in the plane of the sheet. A texture is typical in processed commercially pure aluminum sheet. This texture could explain the presence of special type boundaries which experience little embrittlement. Attempts to measure the texture of these specimens with an x-ray goniometer were unsuccessful due to the large grain size in the specimens.

The approximately 2-D microstructures produced in silicon-iron are quite ductile. Of course, the basic ductility of the materials facilitates rolling operations to produce thin sheet samples. Brittleness has been introduced by the introduction of sulfur into the specimens at temperatures between 600°C and 800°C. At these temperatures, sulfur segregates strongly to the grain boundaries, lowering grain boundary cohesive forces and resulting in a shift from ductile, transgranular failure under stress to brittle, intergranular failure under similar loading conditions. In some cases, a grain boundary phase of iron sulfide can be introduced which further reduces specimen ductility. This material exhibits almost profound brittleness, with little or no resolvable plastic deformation prior to plastic deformation. A variable ratio H_2/H_2S gas flow provides the sulfur and also allows control over the equilibrium concentration of sulfur in the bulk at the treatment temperature. The concentration of sulfur at the grain boundaries, and thus the extent of embrittlement, is controlled by the ratio of gases and the treatment temperature. A full range of toughnesses ranging from complete ductile to complete brittle can be achieved in the 2-D Si-Fe specimens.

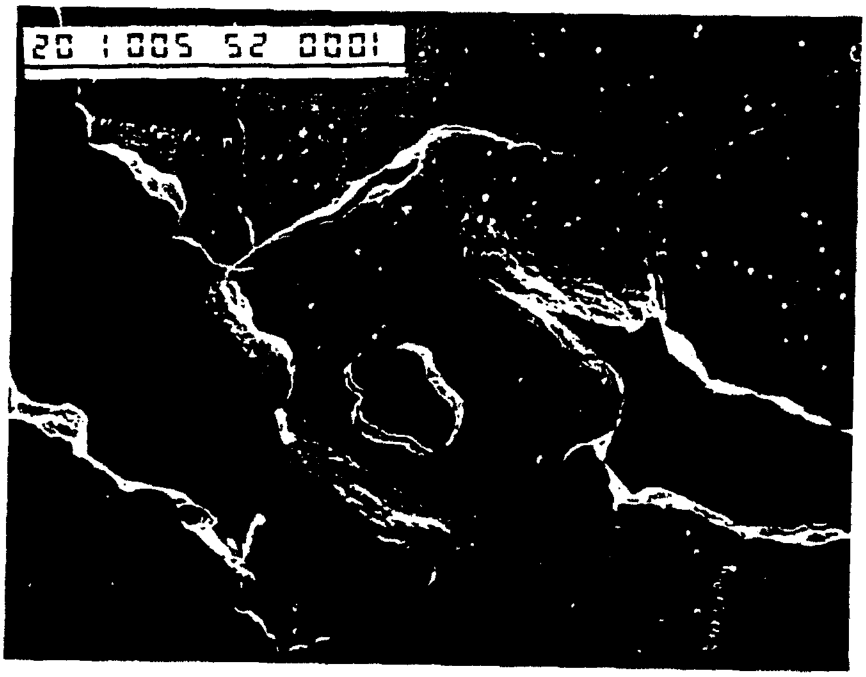

Fig. 7. SEM micrograph of a bridging grain in a biaxially loaded strip of embrittled aluminum with a 2D microstructure.

2.4.2. Oriented Composites

By introducing preferred orientation into composite ceramics, dramatic property anisotropies can be produced. Evaluating extremes of anisotropy in microstructure-property relations can be explored over a range or conditions. To demonstrate anisotropy, preferred orientation and quantitative descriptions of the preferred orientation are essential. Professor Bowman's research group at Purdue has established strong leadership in preferred orientation measurements of high temperature ceramic and intermetallic materials [48-58]. These concepts can be extended into consideration of model materials which exaggerate property differences and thereby enhance anisotropy.

The Masters research of Carl Nogaj was focused on the production of ionic matrix, covalent reinforcement composites wherein the property differences between the matrix and the reinforcement are substantial as shown in Table I. In this work SiC platelet reinforcements in the ionic solid lithium fluoride are used to demonstrate platelet reorientation during hot-forging, suppression of matrix phase texture and property anisotropy in the resulting composites.

Table 1

	Young's Modulus (GPa)	Resistivity (Ω .m)
SiC	400	1-10 Ω.m
Lif	110	>10^5 Ω.m

(Room Temperature)

The schematic figure and micrograph of one of these composites show the essentially planar array of platelets within the composite (Figure 8). These

materials are produced by conventional hot pressing of the model composites at @600°C. followed by hot forging at lower temperatures, typically 400°C. Besides the advantage of large differences in the elastic and electrical properties of the matrix and reinforcement these materials also several other unique characteristics when compared to conventional metal or ceramic matrix composites:

1. Unlike metal matrix composites, the insulating phase is the matrix phase.
2. Processing and deformation can be performed at a relatively low temperature on a material which is essentially brittle at room temperature.
3. Very little chemical reaction occurs between the matrix and reinforcement under these processing conditions.

2.5. Evaluating Damage in Model Microstructures

Another stage of the research is testing the embrittled 2-D specimens in both uniaxial and biaxial tension. In the case of supported tensile tests, specimens are attached to a ductile substrate with adhesive. We have observed the progressive intensification of deformation at grain boundaries in ductile specimens, as well as the progressive failure of brittle specimens [3,19]. 2-D samples have also been tested in an unsupported manner by pulling small 'dog-bone' type specimens cut from the 2-D sheet materials and fracture in the biaxial test rig shown in Figure 4. In sufficiently embrittled materials the failures are completely intergranular in nature. Data obtained on crack initiation and propagation in relation to microstructural aspects will be analyzed in terms of the theoretical models. Using characterization information on the precise intergrain

orientation relationships along with microstructural observations, the relationship between failed edges in supported thin films of two-dimensional materials can be determined. By evaluating crack characteristics including inclination and length the relative fracture resistance of different grain boundary types should be obtainable. This is precisely the type of information inferred by Watanabe and co-workers [2].

2.6. Measuring Constitutive Properties: Elasticity and Conductivity

We have initiated work on the measurement of elastic and electrical conductivity anisotropy in monolithic and composite ceramic materials. These experiments are intended to provide baseline information on the effective properties of materials as a function of microstructural detail. Information on these and other tensor-based properties are necessary to enable later application of nondestructive evaluation techniques to damaged structures. The attenuation of wave propagation, percolation effects in composite materials and the influence of microcracking versus microporosity are all issues which must be addressed to provide a useful format for damage evaluation.

The effective mechanical and physical properties of macroscopic structures composed of discrete microscopic constituents such as individual matrix crystals or fiber platelet or whisker composite reinforcements is a sufficiently difficult problem when each of the constituents is itself isotropic. Under such circumstances, the volume fraction, site distribution, orientation distribution and the shape distribution of the reinforcements and the nature of their bonding to the matrix are the only necessary considerations. Representation of such features, and any possible interactions between the features, i.e. certain sizes or shapes may have a tendency for particular orientations, provides a daunting picture for

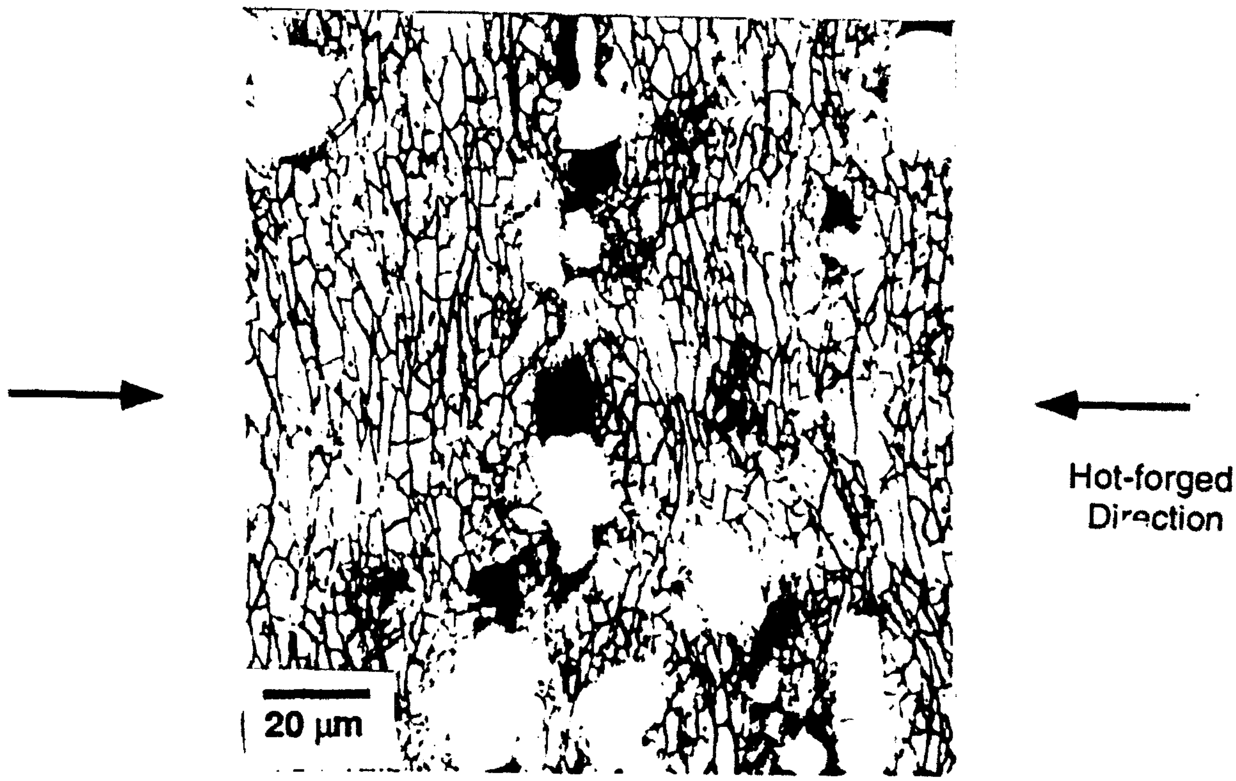

Fig. 8. Hot-forged lithium fluoride composite T = 400°C, $\varepsilon_{T(Plastic)}$ = -0.8.

prediction of mechanical or physical response variability for specimens which may vary in size.

To some extent the scale dependence of properties is dependent on the nature of the property itself. Possibly the best approach is to consider the responsiveness of several properties to the same characteristic. If we imagine the case of a small, idealized matrix perturbation in such as a spherical hole in an otherwise homogeneous material it is clear that the size scale of any measured property, for example the wavelength of a wave passing through the material will determine the effects of the defect on a measured mechanical or physical response. Once the defect is distorted from the spherical case, the orientation of the defect with respect to the direction at which a property is measured becomes important. If the defect is instead a reinforcement phase, with its own physical and mechanical properties, in addition to the shape, the bonding between the matrix and reinforcement will affect mechanical or physical responses. As well, any anisotropy of the property characteristics of the reinforcement will be reflected in the macroscopic response.

For the case of single phase polycrystalline materials which possess both macroscale and microscale anisotropy, texture or preferred orientations of individual crystals Bunge has demonstrated that the probabilistic nature of texture data does provide a clear indication of trends inherent to mechanical or physical properties which can be expressed in a tensorial manner [59]. The accuracy of theoretical fits relies in many cases on the assumptions. Possibly the clearest indication of problems in this technique are shown by application to elastic properties. The two equivalent equations

$$\sigma_{ij} = C_{ijkl}\, e_{kl} \quad \text{and} \quad e_{ij} = S_{ijkl}\, \sigma_{kl}$$

for the respective rank tensors of stiffnesses and compliances can be individually applied to measured properties of crystals and to macroscopic components with defined symmetry axes.

In classical approaches to elasticity of polycrystals the Voigt-Reuss approximations serve as the upper and lower bounds. Although Hill approximated the elastic constants via use of an average of the upper and lower bounds, his approximation has no fundamental foundation. Despite this, approximation of macroscopic anisotropy in materials with oriented microstructures has been fairly successful for predictions of elastic constants in some rolled metals. Kröner and others have proposed self-consistent approaches for determining elastic constants wherein the Eshelby approach of an embedded crystal within a homogeneous matrix can be approached to determine the elastic constants of oriented polycrystals [see 60]. Application of these approaches to specific microstructures, particularly two-phase composites have not been given previously. The models under development by Ostoja should supersede these models by permitting stochastic variability and size-dependence. Unfortunately, very little experimental data correlating texture, microstructure and anisotropy is available.

We point out here that the graph representation of a material microstructure together with the method of neighborhoods outlined above can be used to calculate statistics (averages and scatter) of various much more complicated models. Thus, our future work will encompass materials with:

- irregular microscale geometry, (like the Voronoi of Fig. 1(b))
- local anisotropy,
- nonlinear elastic and possibly dissipative behavior,
- inner- and intra-granular damage distributions,
- correlations in property assignments between nearest and further neighbors on the graph

To provide a convenient materials system for evaluation of effective elastic constants composites in a model ceramic system comprising a lithium fluoride matrix material with a silicon carbide platelet reinforcement have been prepared as discussed above [12]. By producing such materials which provide preferential orientations of the platelet reinforcement phase, it is possible to fabricate a material with different properties within the plane of preferential orientation as opposed to normal to it. Under this controlled microstructural condition and with significant differences in the matrix and reinforcement phase elastic properties, it should be possible to produce sufficient anisotropy for analysis.

The ratio of Young's moduli for LiF/SiC composites is on the order of eight to ten which coupled with strong preferred orientations of the type shown in Figure 4, should provide significant information on effective elastic properties in this model composite. Results on this composite [12] show:

1. Matrix textures of LiF [50] produced by forging are suppressed by platelet additions. This should inhibit matrix effects on elastic anisotropy.

2. The preferred orientation of the platelets is very strong even with moderate forging strains.

The room temperature electrical conductivities of LiF and SiC are dramatically different. This should permit reasonable evaluations of models which have previously been applied to thermal conductivity in composite materials [51]. We have constructed a device which should permit evaluation of the bulk electrical conductivities of model LiF/SiC composites and structural materials which are under investigation in related work at Purdue [51,54].

2.7. Plastic Deformation in Model Materials: Strength and Hardness

To provide baseline information on stochastic property variations we are initiating a series of measurements of hardness wherein the relationship between hardness indentor size and grain size will be varied independently. Several students will be involved in a series of exhaustive studies to determine the variability in hardness measurement and hardness of materials which are considered uniform. We will then attempt to assess the nature of variability as it applies to both sample and microstructure scale. The variability in these measurements will be compared to the recent extensions of stochastic models by Ostoja into evaluation of plastic deformation [15,67].

2.8. Microstructural Effects in Wave Propagation

Dynamic response of microstructures was the final topic studied in our research program. Specifically, we focused on a following problem: *what are the effects of microstructural disorder on the propagation of wavefronts in solid materials?* We pursued this goal in two specific areas - transient waves in granular-type media, and acceleration wave to shock wave transition in white-noise continuous random media.

In the first area a stochastic method was developed [61,62] to study response, due to external impact, of one-dimensional random nonlinear microstructures with material randomness, of high signal-to-noise ratio, being present in physical parameters and grain lengths. It generalizes the classical solution techniques [63], based on the theory of characteristics, by taking advantage of the Markov property of the forward propagating disturbances. Consequently, the magnitude and location in space-time of any disturbance is described by a vector Markov diffusion process. While our work was mainly focused on bilinear media problems, we are now focusing on solutions to

transient wave problems in nonlinear elastic, and linear-hysteretic media, as well as on an extension of our stochastic method to problems of spherical and cylindrical symmetry. In this latter category the main interest is in the effects of material randomness on the waves of unloading vis-a-vis classical solutions for homogeneous media such as developed in [61].

The problem of transition of acceleration waves into shock waves has, in the past, been studied in the setting of deterministic media only [64,65]. In [62,66], which represent a first-ever stochastic study of the subject, we analyzed this problem in the setting of white-noise random media. The problem of shock formation, which involves a stochastic competition of dissipation and elastic nonlinearity, is treated using a diffusion formulation for the Markov process of the inverse emplitude. Our focus has been on the random critical amplitude and the random time (distance) to form a shock. This subject has been followed in [17,18], where the first four moments of the critical inverse amplitude are derived explicitly as functions of the means and crosscorrelations of the underlying vector random process. It is found that the Stratonovich as well as the Ito interpretation of the equation governing the evolution of an acceleration wave lead to an increase of the average critical amplitude of the random medium problem over the critical amplitude of the deterministic homogeneous medium problem. Probability distribution of the critical inverse amplitude is found to be, in general, of Pearson's Type IV. These results are a stepping-stone for extension of these analyses to more realistic (i.e. non-white) media models.

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66. M. Ostoja-Starzewski, "On Wavefront Propagation in Random Nonlinear Media," in *Computational Stochastic Mechanics* (P.D. Spanos and C.A. Brebbia, Eds.), CMP-Elsevier, pp. 687-698 (1991).

67. M. Ostoja-Starzewski, "Plastic Flow of Random Media: Micromechanics, Markov Property and Slip-Lines," *Applied Mechanics Reviews* (Special Issue: *Material Instabilities*), Vol. 45 (3, Part 2), pp. S75-S82 (1992).

68. M. Ostoja-Starzewski, "Wavefront Propagation in Random Granular Media," Session on Wave Propagation in Random Media, *Proceedings of ASCE Specialty Conference on Probabilistic Mechanics and Structural and Geotechnical Reliability*, pp. 384-387, Denver, CO (1992).

69. M. Ostoja-Starzewski and R. Setyabudhy, "Limit Analysis of a Cylindrical Tube of a Random Inhomogeneous Plastic Material under Internal Pressure," 1992 ASME Winter Annual Meeting, in *Recent Advances in Structural Mechanics* (Y. W. Kwon and H. H. Chung, Eds.), PVP-Vol. 248 and NE-Vol. 10, pp. 87-92.

10. M. Ostoja-Starzewski, "Micromechanics as a Basis of Random Elastic Continuum Approximations," *Probabilistic Engineering Mechanics*, in press, 1993.

11. M. Ostoja-Starzewski, "On the Critical Amplitudes of Acceleration Wave to Shock Wave Transition in White-Noise Random Media," *Journal of Applied Mathematics and Physics (ZAMP)*, in press, 1993.

12. K. L. Kruger, C. D. Nogaj and K. J. Bowman, "Texture in Hot-Forged LiF and LiF/SiC Platelet Composites," to be submitted to *Texture and Microstructures*.

13. M. Ostoja-Starzewski, "Micromechanics as a Basis of Stochastic Finite Elements and Differences - an Overview," invited paper, *Appl. Mech. Rev.* (Special Issue: *Mechanics Pan-America 1993*), to appear.

B. Books or Book Chapters Published

1. M. Ostoja-Starzewski, "Percolation Models as a Basis of Material Failure," 4th IUTAM Symposium on *Creep in Structures* (M. Zyczkowski, Ed.), pp. 425-432, Springer-Verlag (1991).

2. M. Ostoja-Starzewski, "On Wavefront Propagation in Random Nonlinear Media," in *Computational Stochastic Mechanics* (P.D. Spanos and C.A. Brebbia, Eds.), CMP-Elsevier, pp. 687-698 (1991).

3. M. Ostoja-Starzewski, "Stochastic Constitutive Laws for Graph-Representable Microstructures," in *Constitutive Laws for Engineering Materials* (C.S. Desai, E. Kremple, G. Frantziskonis, H. Saadatmanesh, Eds.), ASME Press, pp. 457-460 (1991).

4. M. Ostoja-Starzewski, "Percolation, Fractals, and Entropy of Disorder in Damage Phenomena," in *Continuum Models and Discrete Systems*, Vol. 2 (G.A. Maugin, Ed.), pp. 203-210, LONGMAN Scientific and Technical (1991).

5. M. Ostoja-Starzewski, "Transient Waves in a Class of Random Heterogeneous Media," *Proceedings of 2nd Pan American Congress of Applied Mechanics*, Valparaiso, Chile, pp. 764-767 (1991). (Selected among the best papers at the Congress, and invited as A.6.)

6. M. Ostoja-Starzewski, "Boundaiy Value Problems in Plastic Flow of Random Media," ASME Summer Mechanics and Materials Conference, Arizona State University, April/May 1992; in *Plastic Flow and Creep* (H. Zbib, Ed.), AMD-Vol. 135, MD-Vol. 31, pp. 149-158.

7. M. Ostoja-Starzewski, "Random Fields and Processes in Mechanics of Granular Materials," US-Japan Seminar on Micromechanics of Granular Materials, *Studfies in Applied Mechanics*, Vol. 31, pp. 71-80, Elsevier (1992).

8. M. Ostoja-Starzewski, "On a Micromechanical basis of Stochastic Constitutive Laws," Session on Probabilistic Mechanics of Geomaterials, 4 pages in *Proceedings of ASCE Engineering Mechanics Conference*, Texas A&M University (1992).

9. M. Ostoja-Starzewski, "Wavefront Propagation in Random Granular Media," Session on Wave Propagation in Random Media, *Proceedings of ASCE Specialty Conference on Probabilistic Mechanics and Structural and Geotechnical Reliability*, pp. 384-387, Denver, CO (1992).

10. M. Ostoja-Starzewski and R. Setyabudhy, "Limit Analysis of a Cylindrical Tube of a Random Inhomogeneous Plastic Material under Internal Pressure," *1992 ASME Winter Annual Meeting*, in *Recent Advances in Structural Mechanics* (Y.W. Kwon and H.H. Chung, Eds.), PVP-Vol. 248 and NE-Vol. 10, pp. 87-92.

11. M. Ostoja-Starzewski, "Micromechanics as a Basis of Stochastic Finite Elements and Differences," Proceedings of 3rd Pan American Congress of Applied Mechanics, Sao Paulo, Brazil, pp. 427-430, January (1993). (Selected among the best papers at the Congress, and invited as A.13.)

C. Invited Conference Presentations

M. Ostoja:

1. "Markov Random Field Methods for Heterogeneous Materials," Session on Materials Instabilities," 11th U.S. National Congress of Applied Mechanics, Tucson, AZ, May (1990).

2. "How Can Random Fields be Used in the Mechanics of Disordered Media?," Workshop on "Percolation Models of Material Failure," Cornell University, Ithaca, NY, May/June (1990).

3. "Percolation and Morphogenesis of Fractals in a Class of Problems in Solid and Geomechanics," International Conference on the Mechanics, Physics, and Structure of Materials - Aristotle's 23 Centuries Celebration Thessaloniki, Greece, August (1990).

4. "Percolation Models as a Basis of Material Failure," 4th IUTAM Symposium on Creep in Structures, Cracow, Poland, September (1990).

5. "Markov Processes in Wave Propagation in Random Media," First Meeting on Modern Ideas in Mechanics and Related Fields, Portland State University, June (1991).

6. "A Markov Field Method for Plane Plastic Flow of Random Media," Sessions on Material Instabilities, 22nd Midwestern Mechanics Conference, University of Missouri-Rolla, October (1991).

7. "Wavefront Propagation in Nonlinear Random Media," Session on Wave Propagation in Random Media, ASCE Specialty Conference on Probabilistic Mechanics and Structural and Geotechnical Reliability, Denver, CO, June 1992, Proceedings.

8. "Delaunay Networks as Generic Models of Random Microstructures," general invited lecture, 29th Polish Solid Mechanics Conference, Rytro, Poland, September (1992).

K. J. Bowman

1. "Texture of Structural Ceramics," Automotive Materials Conference, Ann Arbor, MI, March (1992).

2. "Texture as Anisotropy in Structural Ceramics," 1992 Ceramic Science and Technology Conference, San Francisco, CA.

3. "Texture and Anisotropy of High Temperature Materials," National Institute of Standards and Technology, Gaithersburg, Maryland, Sept. 25, 1992.

D. Graduate Students

Ostoja::	Khalid Alzebdeh, Mechanics, Ph.D., non-US
	David H. Erwin, Mechanics, Ph.D., US
	Richard Setyabudhy, Mechanics, Ph.D., non-US
	Andys Ierides, Mechanics, M.Sc., non-US
Bowman::	Michael Sandlin, M.S. (1991), US
	Keith Kruger, M.S. (1992), US
	Farnjeng Lee, Ph.D. (1992), non-US
	Yuechu Ma, Ph.D. (1992), non-US
	Carl Nogaj, M.S. (1992), US
	Amy Craft, M.S. (1993), US
	Greg Steinlage, M.S. (1993), US
	Michael Grah, Ph.D. (1993), US

E. External Reports

1. K. J. Bowman, Dept. of Defense, State-of-the-Art Report, "Refractory Metal Disilicides Research."